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Gary M. Hartman Date: 01/12/04

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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.	:	10/063,967	Confirmation No. **4049**
Applicant	:	Yuk-Chiu Lau et al.	
Filed:	:	May 30, 2002	
TC/Art Unit:	:	1762	
Examiner	:	Katherine A. Bareford	
Docket No.	:	17MY-7127	
Customer No.	:	27127	

Commissioner for Patents
P.O. Box 1450
Alexandria VA 22313-1450

DECLARATION UNDER 37 CFR §1.131

Assistant Commissioner for Patents
Washington, D.C. 20231

We, Yuk-chui Lau, Hongyu Wang, and David J. Mitchell, depose and say that:

(1) We are the sole joint inventors of the subject matter covered by each of the claims pending in the above-identified U.S. patent application (the "Application").

Application No. 10/063,967
Docket No. 17MY-7127

(2) We are currently employed as Engineers with the General Electric Company.

(3) The filing date of the Application is May 30, 2002.

(4) Claims 1-25 of the Application are rejected under 35 USC §103 as being unpatentable over EP 1 142 850 A1 to Wang et al. in view of U.S. Patent No. 6,180,184 to Gray et al. (Gray).

(5) EP 1 142850 A1 was published on October 10, 2001.

(6) Prior to October 10, 2001, we had conceived and completed, in this country, our disclosed and claimed invention for a method of depositing a dense, strain-tolerant, vertically-cracked YSZ-containing ceramic layer on a second ceramic layer, as evidenced by the General Electric "Disclosure Letter Outline," Docket No. 17MY-7127, a copy of which is attached hereto as Exhibit A.

(7) Prior to October 10, 2001, we had reduced our invention to practice by depositing YSZ-containing ceramic layers at different temperatures on YSZ/BSAS and YSZ/mullite ceramic layers, whereby deposition (substrate) temperatures capable of avoiding horizontal cracks were established as evidenced by the images of the Figures in the Disclosure Letter Outline.

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I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Yuk-shiu Lam
Chiu Yc

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Hongyu Wang

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

David J. Mitchell

17MY-7127

DISCLOSURE LETTER OUTLINE

Distribution:
Patent Operation
Original & 2 Copies*
via Lab Manager
Immediate Manager
Inventor(s) : Y.C. Lau
Hongyu Wang
David Mitchell

Building: K-1 Room: MB243
Date: [redacted]

Laboratory Manager of each inventor:* Paul Follansbee, PML
Peter Schilke, MPE GEPS
Fred Mazandarany, CL

SUBJECT: PATENT DISCLOSURE LETTER

Processing of Thermal/Environmental Barrier Coatings with Compositional Gradient for Silicon-based Materials.

1. SUMMARY

This invention involves processing techniques and parameters for preparing thermal/environmental barrier coatings on Si-based ceramic or ceramic composite substrates. The invention will enable the use of Si-based materials at high temperature under engine combustion environments.

2. OBJECT OF INVENTION (e.g., problem, opportunity, prior art)

Materials containing silicon, particularly those with silicon carbide (SiC) as a matrix material and/or reinforcing material (such as SiC/SiC ceramic matrix composite (CMC)), are currently being considered for high temperature applications, such as combustor, airfoils, nozzles and other hot section components of gas turbine engines. In many applications, protective coatings over the Si-containing material are required. For example, a thermally insulating layer will reduce the CMC surface temperature and enable the use of CMC under higher temperatures than without the thermal protection. Additionally, such coatings should provide environmental protection by inhibiting the major mechanism for degradation of silicon-containing materials in corrosive environments, i.e., the formation of volatile silicon hydroxide products (mainly $Si(OH)_4$). Other important property of the coating material includes a coefficient of thermal expansion (CTE) compatible with the Si-containing material, low permeability for oxidants, and chemical compatibility with the Si-containing material and silica scale formed from oxidation. The coating essentially has a dual function serving as a thermal barrier and simultaneously providing protection from the environment. A coating system having such dual function is termed a thermal/environmental barrier coating (T/EBC).

This coating system can be produced by air plasma spraying. The microstructure and mechanical integrity of this coating system depends on the processing temperatures. This invention describes the process temperature and parameters that will produce a T/EBC system with desirable mechanical integrity.

3. DESCRIPTION OF INVENTION

A T/EBC coating system recently developed jointly by CRD and GEAE (GEAE Docket #13DV13216) consists of five layers:

1. Si bond layer,
2. Mullite/Barium-Strontium-Aluminosilicate (BSAS) layer as a transition layer to the third layer,
3. BSAS layer providing excellent environmental protection to the Si-based materials,
4. BSAS or mullite/yttria-stabilized zirconia (YSZ) layer as a transition layer to the fifth layer, and
5. YSZ layer as the thermal barrier (insulating) coating due to its low thermal conductivity

This is a complex coating system which requires a well-controlled coating process. A set of coating deposition parameters have been developed previously for environmental barrier coatings (EBC) that consists of the first three layer in the above design. This invention focused on the processing of the fourth and fifth layer.

Since the YSZ has a large CTE (coefficient of thermal expansion) mismatch with Si-based ceramic substrate and other coating constituents, a transition layer (the fourth layer) is needed. The composition (both type and amount of materials) of the fourth layer is important in adjusting CTE and enabling the transitioning function. This invention identified YSZ/mullite (50/50 volumetric mixture) and YSZ/BSAS (50/50 vol%) as the suitable composition.

Also because of the large CTE mismatch of YSZ, the processing parameters, especially those affecting the substrate temperatures, are crucial in achieving the proper microstructure. These parameters include gun power, stand-off distance, gun travelling span, and gas flow rates. This invention has tested and verified a set of desirable processing parameters.

4. OTHER INFORMATION (e.g., test data, reduction to practice, planned use)

This coating system is produced by air plasma spraying. Depending on the deposition temperatures during the application of the YSZ top layer, the microstructure and mechanical integrity of the coating system vary. Figure 1a shows a desirable microstructure of the five-layer T/EBC system when the maximum substrate temperature during the YSZ topcoat deposition is about 450°C. In this case, the 4th transition layer is YSZ/BSAS. The YSZ topcoat microstructure is dense with vertical cracks that are desirable for improved strain-tolerance (U.S. patent #6180184). At a much higher deposition temperatures, e.g., ~550°C, the stress due to thermal expansion mismatch between the various layers is much higher resulting in the formation of large horizontal cracks in the YSZ as well as in the BSAS layers as shown in Fig. 1b. The formation of these horizontal cracks is detrimental to the mechanical integrity of the coating system because they will cause the entire coating system to wrinkle and hence reduce the protective power of the coating system

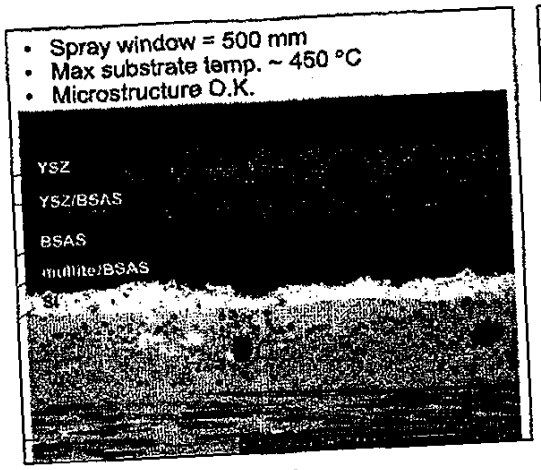

Fig. 1a

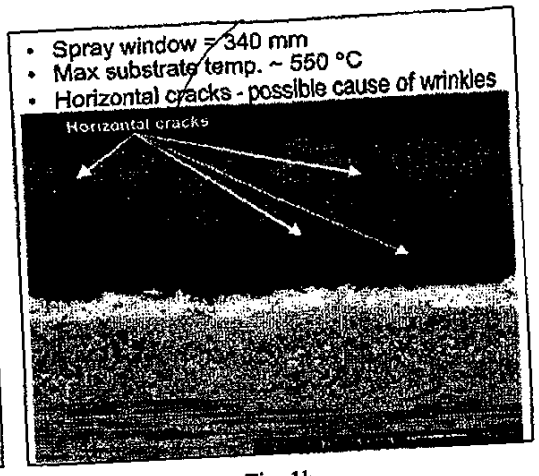

Fig. 1b

Figure 1: T/EBC microstructure @ (a) 450°C and (b) 550°C deposition temperature during the application of YSZ topcoat.

The durability and protective capability of the T/EBC coating system may be examined by testing in a high steam cyclic furnace (HSCF) test, where a coated specimen is exposed to a 90% H_2O/10% O_2 flowing atmosphere. The specimen is cycled from 2400°F in the HSCF to room temperature at regular intervals to produce thermal stresses in the coating.

Figure 2 shows the microstructure of a coated specimen after the HSCF testing. This specimen was coated with a coating system identical to that of Fig. 1b under the same deposition conditions, i.e., maximum deposition temperature of the YSZ top coat was ~550°C. This coated specimen had a wrinkled appearance prior to the HSCF test, and the damage that resulted is apparent when compared with the microstructure shown in Fig. 1b. It is apparent that the wrinkled appearance is the result of horizontal cracking due to relatively high YSZ deposition temperature, which propagates and causes spallation of the coating during thermal cycling.

Figure 3 shows the microstructure of a T/EBC after exposure in the HSCF under the same conditions as those experienced by the specimen shown in Fig. 2. The 4th transition layer of this coating system was YSZ/mullite. Processing of this coating system did not result in a wrinkled appearance, and the coating did not spall during exposure. Figure 3 shows that processing techniques which produce a coating free of horizontal cracks produce a durable coating which does not undergo spallation during thermal cycling.

5. RECORDS

Records for the above reduction-to-practice experiments are kept in forms of plasma spray run sheets and lab notebooks.

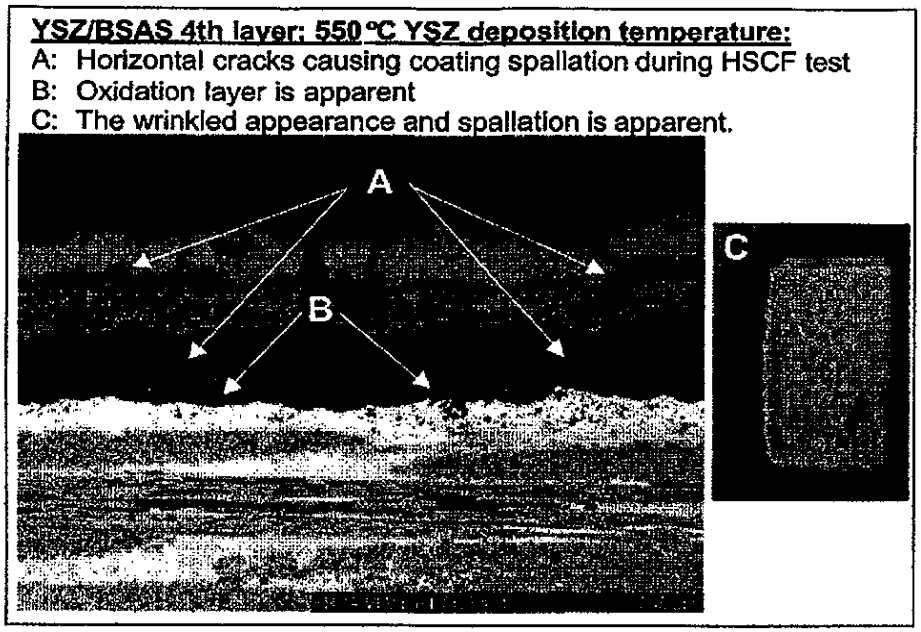

Figure 2: T/EBC microstructure that was wrinkled after exposure for 250 thermal cycles, 500 hours, in a 90% H_2O/10% O_2 flowing atmosphere at 2400°F in the HSCF.

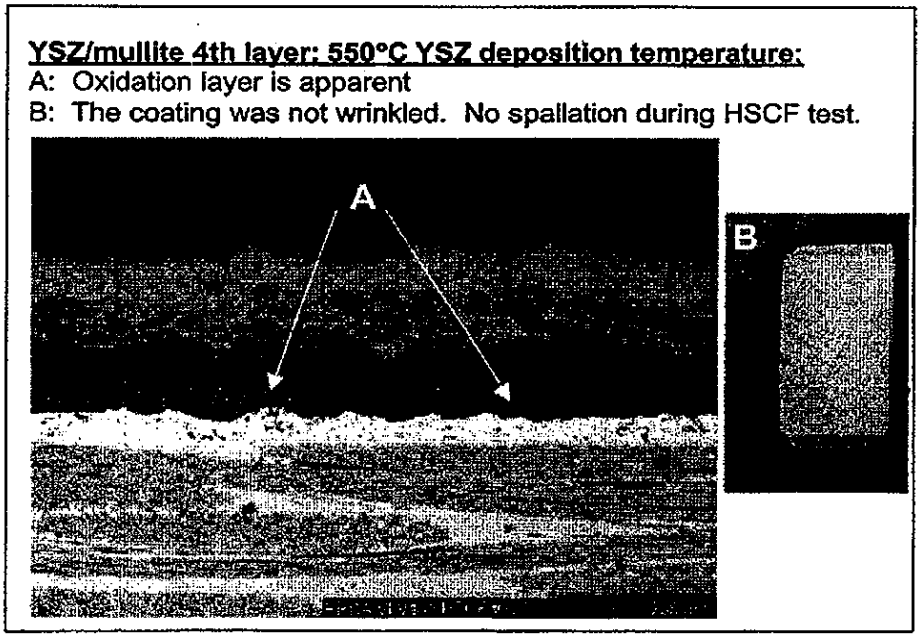

Figure 3: T/EBC microstructure that was not wrinkled after exposure for 250 thermal cycles, 500 hours, in a 90% H_2O/10% O_2 flowing atmosphere at 2400°F in the HSCF.

6. WITNESSES AND DATE

READ AND UNDERSTOOD BY: *INVENTOR: _____
Signature

WITNESS: _____ Yuk-chiu Lau
Signature Type Inventor's Name

Date: _____ GECRD PML
Laboratory or Program

Date: _____

READ AND UNDERSTOOD BY: *INVENTOR: _____
Signature

WITNESS: _____ Hongyu Wang
Signature Type Inventor's Name

Date: _____ GEPS M&PE
Laboratory or Program

Date: _____

*INVENTOR: _____
Signature

David Mitchell
Type Inventor's Name

GECRD CL
Laboratory or Program

***When the invention is joint, all inventors should sign and date the disclosure letter in the order they would like their names to appear on the issued patent.**

(Complete and attach an Invention Disclosure Statement, Form RD-506A, for each inventor.)